



PATENT  
Docket No.: 15275/8611 (Dobbins 2-1)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Reissue Application No. : 08/833,620 )  
Filed : April 7, 1997 ) ) Examiner: J. Hoffman  
U.S. Patent No. : 5,043,002 ) )  
Granted : August 27, 1991 ) ) Art Unit: 1731  
Patentees : Michael S. Dobbins )  
              Robert E. McLay )  
For : METHOD OF MAKING FUSED SILICA )  
      BY DECOMPOSING SILOXANES )

U.S. Patent and Trademark Office  
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Sir:

Transmitted herewith in the above-identified application are:

- Appeal Brief (18 pages) with Appendix (7 pages) (in triplicate);
- Request for Two-Month Extension of Time;
- A check in the amount of \$720.00 to cover the appeal fee and the two-month extension of time fee; and
- A self-addressed, prepaid postcard for acknowledging receipt.

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Date: December 23, 2002

  
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(143)

APPEAL BRIEF

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Dear Sir:

Pursuant to 37 CFR § 1.192, appellants hereby file their appeal brief in triplicate. Enclosed is the filing fee of \$320.00 required by 37 CFR § 1.17(c). The Commissioner is hereby authorized to charge/credit Deposit Account No. 14-1138 for any deficiency/overage.

**I. REAL PARTY IN INTEREST**

Corning Incorporated, as assignee of U.S. Reissue Patent Application No. 08/833,620, is the real party in interest.

**II. RELATED APPEALS AND INTERFERENCES**

There are no related appeals or interferences pertaining to the above-identified application.

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### III. STATUS OF CLAIMS

A. Claims 12, 13, 22, 33-39, 41-44, 46, 47, 49, and 51-53 Are Finally Rejected

Claims 12, 13, 22, 33-39, 41-44, 46, 47, 49, and 51-53 have been finally rejected under 35 U.S.C. § 103(a) for obviousness over U.S. Patent No. 4,501,602 to Miller et al. (“Miller”) in view of European Patent No. 38,900 to Schwarz et al. (“Schwarz”) and, optionally, U.S. Patent No. 2,272,342 to Hyde (“Hyde”) and/or Japanese Kokai Patent Application No. 138145 to Kawaguchi et al. (“Kawaguchi”).

B. Claims 1-11, 14-21, 23-32, 40, and 50 Have Been Canceled

Claims 1-11, 14-21, 23-32, 40, and 50 have been canceled.

C. Claims 45 and 48 Stand Allowed

Claims 45 and 48 are allowed.

D. Claims 12, 13, 22, 33-39, 41-44, 46, 47, 49, and 51-53 Are On Appeal

The decision of the examiner finally rejecting claims 12, 13, 22, 33-39, 41-44, 46, 47, 49, and 51-53 is appealed. These claims, in their currently pending form, are set forth in the attached Appendix.

### IV. STATUS OF AMENDMENTS

There are no amendments pending.

## V. SUMMARY OF INVENTION

### A. Problems In The Prior Art

Various processes are known in the art that involve the production of metal oxides from vaporous reactants (column 1, lines 6-7 of U.S. Patent No. 5,043,002). The most basic requirements of such processes necessitate a feedstock solution, a means of generating and transporting vapors of the feedstock solution (hereafter called vaporous reactants) and an oxidant to a reaction site, and a means of catalyzing oxidation and combustion coincidentally, producing finely divided, spherical aggregates, called soot (column 1, lines 8-14). This soot may be collected and simultaneously or subsequently heat treated to form a non-porous, transparent, high purity glass article (column 1, lines 14-18). The means for executing these reactions is usually a specialized piece of equipment with a unique arrangement of nozzles and burners (column 1, lines 18-20).

Much of the initial research that led to the development, and thus patent protection, of a plethora of such processes focused on the production of fused silica (column 1, lines 21-23). Selection of the appropriate feedstock was found to be as important in the production of high purity fused silica as the equipment used in its production (column 1, lines 24-26). Consequently, a material was identified that could generate the needed vapor pressure of 200-300 mm at temperatures below 100°C; the high vapor pressure of silicon tetrachloride (SiCl<sub>4</sub>) isolated it as a convenient vapor source for soot generation, thus launching the discovery and use of a series of similar chloride-based feedstocks (column 1, lines 26-32). This factor, more than any other, is responsible for the presently accepted use of SiCl<sub>4</sub>, GeCl<sub>4</sub>, POCl<sub>3</sub>, and BCl<sub>3</sub> as vapor sources, even though these materials have certain chemically undesirable properties (column 1, lines 32-36).

Silicon, germanium, zirconium, and titanium are metals often used in halide form as vaporous reactants for forming high purity metal oxide glasses (column 1, lines 37-39). However, SiCl<sub>4</sub> has been the industry standard among metal-source vaporous reactants used over the years for the production of high purity silica glasses (column 1, lines 39-42). As disclosed in U.S. Pat. No. 3,698,936, one of several

reactions may be employed to produce high purity fused silica via oxidation of  $\text{SiCl}_4$ ; namely:

- (1)  $\text{SiCl}_4 + \text{O}_2 \rightarrow \text{SiO}_2 + \text{Cl}_2$ ,
- (2)  $\text{SiCl}_4 + \text{O}_3 \rightarrow \text{SiO}_2 + \text{Cl}_2$ , or
- (3)  $\text{SiCl}_4 + \text{H}_2\text{O} \rightarrow \text{SiO}_2 + \text{HCl}$ ,

whereby burners or jet assemblies are utilized in feeding the reactant gases and vapors to a reaction space (column 1, lines 42-50). There are inherent economic disadvantages to each of these reactions (column 1, lines 50-52).

These reactions, which oxidize  $\text{SiCl}_4$  through pyrolysis and hydrolysis, have the disadvantage of producing a very strong acid by-product (column 1, lines 53-55). While the first two reactions occur theoretically, it is likely that an auxiliary fuel is needed to achieve pyrolytic temperature, thus leading to hydrolysis of the silicon tetrachloride and formation of hydrochloric acid (HCl) (column 1, lines 55-59). Such a by-product is not only a detriment to many deposition substrates and the reaction equipment, but also is a detriment to the environment (column 1, lines 59-62). Emission abatement systems have proven to be very expensive due to down-time, loss, and maintenance of equipment caused by the corrosiveness of HCl (column 1, lines 62-65).

The first reaction, which utilizes oxygen as it occurs naturally, requires elevated reaction temperatures which, generally, are difficult to maintain without using specialized equipment (column 1, lines 66 to column 2, line 1). The second reaction requires ozone, an unstable form of molecular oxygen that not only warrants special handling, but also must be manufactured on site due to a lack of commercial availability (column 2, lines 1-4). Notwithstanding the handling and disposal of the HCl by-product necessitated by the hydrolysis and pyrolysis of  $\text{SiCl}_4$ , the third reaction, also hydrolysis of  $\text{SiCl}_4$ , tends to be the preferred commercial method of producing fused silica for economic reasons (column 2, lines 5-8).

Though hydrolysis of  $\text{SiCl}_4$  has been the preference of industry for producing high purity fused silica over the years, the enhanced global sensitivity to environmental protection has led to more strict government regulation of point source emissions, prompting a search for less environmentally pernicious feedstocks (column

2, lines 10-15). In new point source emission regulations, HCl, the by-product of hydrolyzing SiCl<sub>4</sub>, as well as many particulate pollutants, has to be cleansed from exhaust gases prior to their release into the atmosphere (column 2, lines 16-19). The economic consequences of meeting these regulations have made commercial production of fused silica by downstream removal of HCl and other metal oxides from halide-based feedstocks less attractive to industry (column 2, lines 19-23).

As an alternative, high purity fused quartz or silica may also be produced by thermal decomposition and oxidation of silane, a compound that requires taking safety measures in handling due to the violent reaction caused when air is introduced into a closed container of silane (column 2, lines 24-29). Silane is commonly reacted with carbon dioxide, nitrous oxide, oxygen, or water to produce a high purity material that is useful in producing, among other things, semiconductor devices (column 2, lines 29-32). However, silane has proven to be much too expensive and reactive to be considered for commercial use except possibly for extremely high purity applications (column 2, lines 32-35).

**B. Brief Description Of The Invention**

The instant invention utilizes halide-free, silicon-containing compounds as a replacement for the halide-based source feedstocks that are often oxidized by flame hydrolysis or pyrolysis, to produce transparent, high-purity silica glass articles (column 3, lines 3-7 of U.S. Patent No. 5,043,002). Fused silica glass produced through the use of silicon-containing compounds as the feedstock components results in carbon dioxide and water as by-products (column 3, lines 7-10). Appellants have found that polymethylcyclosiloxanes are particularly useful as substitutes for halide-based, silicon-containing compounds (column 3, lines 10-14). Hexamethylcyclotrisiloxane (HMCTS), octamethylcyclotetrasiloxane (OMCTS), and decamethylcyclopentasiloxane (DMCPS) are representative of operable polymethylcyclosiloxanes (column 3, lines 14-19). OMCTS and DMCPS have been found to be the most preferred (column 3, lines 19-20).

It will be appreciated that, as in the current commercial processes for doping fused  $\text{SiO}_2$  articles produced via the hydrolysis/oxidation of  $\text{SiCl}_4$  with various metals in order to modify the chemical and/or physical properties thereof, the fused  $\text{SiO}_2$  articles prepared in accordance with the present invention can likewise be doped with metals (column 3, lines 35-41). For example, fused  $\text{SiO}_2$  articles have been doped commercially with  $\text{Al}_2\text{O}_3$ ,  $\text{B}_2\text{O}_3$ ,  $\text{GeO}_2$ ,  $\text{P}_2\text{O}_5$ , and  $\text{TiO}_2$  utilizing halide-containing compounds of aluminum, boron, germanium, phosphorous, and titanium, respectively (column 3, lines 41-45). Similar dopants can be utilized in the present inventive process but would, of course, provide a source of halide emissions (column 3, lines 45-47). Consequently, to eliminate point source emissions of halides, organometallic compounds of the dopant metals should be employed (column 3, lines 47-50).

In summary, the present invention comprehends doping of fused  $\text{SiO}_2$  articles with  $\text{P}_2\text{O}_5$  and/or at least one metal oxide selected from Groups IA, IB, IIA, IIB, IIIA, IIIB, IVA, IVB, VA, and the rare earth series of the Periodic Table (column 3, line 68 to column 4, line 4).

## VI. ISSUE

Whether claims 12, 13, 22, 33-39, 41-44, 46, 47, 49, and 51-53 are properly rejected under 35 U.S.C. § 103(a) for obviousness over Miller in view of Schwarz and, optionally, Hyde and/or Kawaguchi in the absence of any motivation to combine these references and in view of appellants' evidence of unexpected results.

## VII. GROUPING OF CLAIMS

As set forth in Section VIII(B)(3)(b), dependent claims 36-38 and 51-53 are patentable of their own accord. All other dependent claims stand or fall with the independent claim from which they depend.

## VIII. ARGUMENT

### A. Applicable Law – 35 U.S.C. § 103

35 U.S.C. § 103 imposes the requirement that an invention, to be patentable, must not have been obvious over the prior art “at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains.” The starting point for discussion of obviousness is Graham v. John Deere Co., 383 U.S. 1 (1966), which set forth the following factors for determining obviousness: (1) the scope and content of the prior art; (2) differences between the prior art and the claims at issue; (3) the level of ordinary skill in the pertinent art; and (4) such objective evidence of non-obviousness as commercial success, long felt but unresolved needs, and failure of others. All evidence must be weighed before reaching a conclusion on obviousness under § 103. Panduit Corp. v. Dennison Mfg. Co., 810 F.2d 1561, 1561, 1 USPQ2d 1593, 1594 (Fed. Cir.), cert. denied, 481 U.S. 1052 (1987); Hodosh v. Block Drug, 786 F.2d 1136, 1143, 229 USPQ 182, 188 (Fed. Cir.), cert. denied, 479 U.S. 827 (1986); Simmons Fastener Corp. v. Illinois Tool Works, 739 F.2d 1573, 1575, 222 USPQ 744, 746 (Fed. Cir. 1984), cert. denied, 471 U.S. 1065 (1985). In addition, the prior art itself must suggest the desirability and, therefore, obviousness of a modification of a reference or the combination of references to achieve a claimed invention. Hodosh v. Block Drug, 786 F.2d at 1143 n.5, 229 USPQ at 187 n.5; In re Gordon, 733 F.2d 900, 902, 221 USPQ 1125, 1127 (Fed. Cir. 1984).

### B. The Rejection of Claims 12, 13, 22, 33-39, 41-44, 46, 47, 49, and 51-53 Under 35 U.S.C. § 103(a) for Obviousness Over Miller In View of Schwarz, and, Optionally, Hyde and/or Kawaguchi, Is Improper

#### 1. Description of Miller

Miller relates to producing glass optical waveguides by generating a silica soot, depositing it on a surface, and consolidating the deposited soot to produce a glass. To the extent a source for the silicon soot is specifically identified, Miller mentions silicon tetrachloride (See Example 7). There is no disclosure of polymethylcyclosiloxane.

2. Description Of Schwarz

Schwarz relates to a method of making pyrogenic silica with siloxanes, such as hexamethylcyclotrisiloxane or octamethylcyclotetrasiloxane. Schwarz manufactures a silicic acid dispersion having a specific BET surface of  $250 \pm 25$  to  $350 \pm 25 \text{ m}^2/\text{g}$  and a thickening viscosity of 4000 to 8000 mPas in unsaturated polyesters by burning a siloxane or mixture of siloxanes in the presence of hydrogen or a hydrocarbon. Suitable siloxanes include hexamethylcyclotrisiloxane or octamethylcyclotetrasiloxane.

3. Description of Hyde

Hyde discloses making a transparent article of silica by oxidizing silicon. Like Miller, Hyde contains no disclosure of using polymethylcyclosiloxane. Only the use of silicon chloride, silicon chloroform, methyl silicate, and ethyl silicate, which are very different from polymethylcyclosiloxane, are mentioned in Hyde.

4. Description Of Kawaguchi

Kawaguchi relates to a method of making a quartz glass element by synthesizing silica particles from a silane compound or a siloxane compound (e.g., hexamethyldisiloxane) and depositing the particles on a rotating carrier at a thickness of 1-300  $\mu\text{m}$ . After deposition, the particles on the carrier are vitrified to form a glass. There is no disclosure of preparing glass from a polymethylcyclosiloxane.

5. The Combination of Miller, Schwarz, Hyde, and Kawaguchi Would Not Have Rendered the Claimed Invention Obvious

It is the examiner's position that the method of making fused silica, as disclosed in the present invention, would have been obvious in view of the combination of Miller, Schwarz, Hyde, and Kawaguchi. Miller is cited as teaching the claimed method of making either optical waveguide fibers or a non-porous body, each made of high purity fused silica, in accordance with the present invention, except

that it utilizes silicon tetrachloride as the source of silicon. However, Schwarz is cited as teaching the use of cyclosiloxanes instead of silicon tetrachloride to eliminate the use of chlorine and the production of acids. Hyde is cited to show that one of ordinary skill in the art would expect hydrolyzable compounds to be useful in making silica soot. Further, it is asserted that one of ordinary skill in the art would have expected the use of polymethylcyclosiloxanes to be successful in view of Kawaguchi's use of siloxanes in making high quality silica glass.

A proper *prima facie* showing of obviousness requires the examiner to satisfy three requirements. First, the prior art relied upon, coupled with knowledge generally available to one of ordinary skill in the art, must contain some suggestion which would have motivated the skilled artisan to combine references. See In re Fine, 837 F.2d 1071, 1074, 5 USPQ2d 1596, 1598 (Fed. Cir. 1988). Second, the examiner must show that, at the time the invention was made, the proposed modification had a reasonable expectation of success. See Amgen v. Chugai Pharm. Co., 927 F.2d 1200, 1209, 18 USPQ2d 1016, 1023 (Fed. Cir. 1991). Finally, the combination of references must teach or suggest each and every limitation of the claimed invention. See In re Wilson, 424 F.2d 1382, 1385, 165 USPQ 494, 496 (CCPA 1970).

a.      Schwarz, Kawaguchi, Miller, and Hyde Do Not Establish Even a *Prima Facie* Case of Obviousness

Of the four references cited in support of the examiner's rejection, only Schwarz utilizes polymethylcyclosiloxane. However, Schwarz is only using this material to prepare silicic acid. There is no suggestion in Schwarz that the resulting silicic acid dispersion is suitable for build up as a deposit on a support. There is also no indication that such a deposit can be consolidated to form a consolidated glass body. Indeed, using the procedure of Schwarz in this manner would be contrary to the teachings of this reference which seeks to make a high surface area dispersion that never builds up as a deposit and does not undergo consolidation. There is no expectation that the technique of Schwarz, which processes a polymethylcyclosiloxane to form a silicic acid dispersion, would be useful in conjunction with a process for making a consolidated glass monolith. Since this is, in

fact, what Miller, Hyde, and Kawaguchi are making, one of ordinary skill in the art would have had no reason to combine Schwarz with Miller, Hyde, and Kawaguchi to produce high purity fused silica glass.

The examiner has taken the position that Miller and Hyde disclose the same invention as that claimed by appellants, except that Miller and Hyde do not teach the use of a polymethylcyclosiloxane. Kawaguchi also makes no explicit mention of using this material. It would, therefore, not provide any motivation to use polymethylcyclosiloxanes to prepare glass. Schwarz is the only reference properly relied on as disclosing the claimed polymethylcyclosiloxane. However, appellants submit that the combination of these references does not form a proper basis to reject the claims for obviousness.

Appellants do not dispute that the claimed siloxanes were known at the time the present invention was made. However, this is not the issue. The question is whether it would have been obvious at that time to utilize such cyclic siloxanes to make non-porous silicon dioxide glass by forming  $\text{SiO}_2$  particles, depositing the particles on a support, and consolidating the deposited particles into a non-porous glass body, as claimed. If Schwarz is simply cited as teaching that polycyclosiloxanes were known, then there is no suggestion to use polycyclosiloxanes in the claimed process, let alone any expectation that such materials could be successfully used to produce a non-porous, high purity fused silica glass.

Schwarz fails to teach numerous aspects of the present invention, including depositing silicon dioxide particles on a support and consolidating the deposited particles into a non-porous high purity fused silica glass. Further, the silicic acid dispersion of Schwarz is never built up on a carrier as a deposit and consolidated. It, instead, is produced in a dispersed form and used in this state to thicken a variety of products. There is no suggestion in Schwarz that the resulting silicic acid dispersion is suitable for build up as a deposit on a support. There is also no indication that such a deposit can be consolidated to form a consolidated glass body. Indeed, using the procedure of Schwarz in this manner would be contrary to the teachings of this reference which seek to make a high surface area dispersion that never builds up as a deposit and does not undergo consolidation. There is no basis to expect that the technique of Schwarz, which processes a polymethylcyclosiloxane to

form a silicic acid dispersion, would be useful in conjunction with a process for making a consolidated glass monolith. Since this is, in fact, what Kawaguchi, Miller, and Hyde are making, one of ordinary skill in the art would have no reason to combine Schwarz with Miller, Hyde, and Kawaguchi.

At the time that the present invention was made, researchers in the art would not have expected the polycyclosiloxanes of Schwarz to be useful in producing a non-porous body of high purity fused silica glass (See the Declaration of Michael S. Dobbins Under 37 CFR § 1.132 ("Dobbins Declaration") ¶ 5, submitted with Supplemental Amendment Under 37 CFR § 1.116, dated November 23, 1999). In particular, Schwarz's polycyclosiloxanes have a large number of carbon atoms and not all of these carbon atoms would have been expected to combust when passed through a burner (*Id.*). Indeed, the production of carbon when burning polycyclosiloxanes to form pyrogenic or fume silica or silicic acid is well documented in the literature (*Id.*). GB 2,049,641 to Kratel et. al., (page 1, lines 6 to 28) states the following:

Very finely divided silica (highly disperse silica) may be manufactured by flame hydrolysis, which comprises reacting a gaseous or vaporisable silicon compound and, optionally, another gas that will burn to form water, with oxygen in a flame (see, for example, DE 900 339, U.S. 2,399,687, G.B. 17325/77 Serial No. 1562966 (equivalent to DE 26 20 737 A1)). Silica manufactured in this manner is known as pyrogenic silica or fume silica. Satisfactory results can be obtained by this method when silicon tetrachloride is used as the gaseous silicon compound. It is, however, often advantageous to use an organosilane as the gaseous silicon compound, but the silica produced from these compounds tends to be contaminated with carbon and thus tends to be dark in colour. This result when using silicon compounds containing silicon-bonded organic groups, especially halogen-containing silicon compounds, has previously been counteracted by using an additional fuel, namely an additional gas that will burn to form water, for example hydrogen or hydrocarbon.

*PCV Siloxane*  
*an*

(Id.). In addition, J. Lipowitz, "Flammability of Poly(Dimethylsiloxanes). 1. A Model for Combustion," J. Fire & Flammability 7: 482-503 (1976) states:

Under fuel-rich conditions ( $\phi > 1$ ), two-stage combustion is evident by formation of inner and outer flame cones. The outer flame cone is a pale bluish color typical of the outer cone of organic flames, both representing further combustion of CO and H<sub>2</sub>. However, the inner cone is blue at  $\phi < 2.67$  and luminous yellow-white at  $\phi > 2.67$ . Organic flames are luminous yellow due to blackbody radiation from carbon particles. Significantly, adiabatic calculations show carbon formation from D<sub>4</sub> and MM at  $\phi > 2.67$ .

\* \* \*

Gray-brown amorphous silica collected above diffusion flames, which are luminous (yellow-white), contains 3-4% total C, primarily elemental carbon, and 0.1-0.2% H. A weak infrared band indicating some C-H bond structure is present at 2925 cm<sup>-1</sup>. No crystallinity indicative of SiO<sub>2(g)</sub>, graphite, SiC, or Si<sub>3</sub>N<sub>4</sub> is found by x-ray powder diffraction or electron diffraction. Detectability limits are estimated at several %. Particulates consist of 100Å diameter particles tightly aggregated into 700-1000Å clumps which are further agglomerated in chains (Figure 5). Some larger particles (2000-4000Å diameter) appear to be carbon and should contribute appreciably to the observed luminosity.

(Id.).

When, in accordance with the teachings of Schwarz, the dispersion product is used as a thickener, the existence of carbon in the dispersion would not present any difficulties (Dobbins Declaration ¶ 6). In fact, the presence of carbon would most likely have improved thickening (Id.).

However, the presence of carbon impurities could result in significant problems when making a non-porous body of high purity fused silica glass, particularly where that glass is used to make precision optical products like optical waveguide fibers (Dobbins Declaration ¶ 7). In making a non-porous body of high purity fused silica glass, scientists skilled in this area would not want any carbon to be present, not even at a parts per million level (Id.). Their concern at the time inventor

Michael S. Dobbins made his invention would have been that production of carbon during glass formation could adversely affect light transmission in a number of ways (Id.). In particular, the presence of carbon particles in the glass would absorb light and cause light scattering resulting in transmission losses (Id.). Moreover, carbon particles in the glass would also be likely to associate with adjacent oxygen atoms that otherwise form silicon dioxide, resulting in C-O bond formation within the glass (Id.). Such C-O bonds would absorb light in the infrared region of the spectrum and cause transmission losses which prevent the light from traveling as far (Id.). This is a particularly significant problem in optical fibers where transmission is in the infrared region of the spectrum and such light must travel long distances (Id.). Carbon formation would also have been expected to physically remove oxygen (otherwise in the form of silicon dioxide within the glass) away from the glass as carbon monoxide (Id.). This would result in an electron deficiency in the resulting glass that would cause transmission loss in the ultraviolet region of the spectrum (Id.). Thus, the formation of carbon in a non-porous body of high purity fused silica glass, such as that used in optical fibers or precision lenses, would be highly undesirable (Id.).

By contrast, all of the claims of the present application call for the production of a non-porous high purity fused silica glass, while claims 13, 22, and 46 set forth methods of making optical waveguide fibers. In view of the recognition in the art that passing polycyclosiloxanes through the flame of a burner would have been expected to produce carbon, scientists making a non-porous body of high purity fused silica glass would not have wanted to make such products by burning polycyclosiloxanes (Id.). In rejecting the claims over the combination of Miller, Schwarz, Hyde, and Kawaguchi, it is exactly this use of Schwarz's polycyclosiloxanes which is proposed.

In response to the above points raised in the Dobbins Declaration, the examiner refers to paragraph 7 of the Declaration of Dale R. Powers under 37 C.F.R. § 1.132 ("Powers Declaration"), which accompanied the June 30, 2000, Request for Reconsideration. In particular, the Powers Declaration, in addition to comparing silica deposition rates for octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, and hexamethyldisiloxane at equivalent flowrates, attempts to achieve an equivalent atomic Si delivery rate for hexamethyldisiloxane by

increasing its flow rate. The effort was unsuccessful. However, it has nothing to do with the Dobbins Declaration which relates to the skilled artisan's lack of motivation to utilize polymethylcyclosiloxanes to make fused silica glass for fear of generating excessive amounts of carbon.

b. The Evidence of Unexpected Results Rebuts Any *Prima Facie* Case of Obviousness

Even if, assuming *arguendo*, the combination of Miller, Schwarz, Hyde, and Kawaguchi could be properly used to reject the claimed invention, which it cannot, any *prima facie* case of obviousness would be rebutted by the evidence of unexpected results achieved by the present invention. See In re De Blauwe, 736 F.2d 699, 222 USPQ 191 (Fed. Cir. 1984).

In particular, evidence of unexpected results is found in the Powers Declaration which shows that the results achieved when utilizing a polymethylcyclosiloxane are substantially better than when a linear siloxane is used. More particularly, a series of tests were conducted under similar conditions (including a siloxane volumetric flow rate of 10 cc/minute) to evaluate the production and deposition rates of silica soot produced from vaporized octamethylcyclotetrasiloxane, hexamethyldisiloxane, and decamethylcyclopentasiloxane (Powers Declaration ¶¶ 4-5). These tests showed that the above siloxanes produced the following amounts and rates of silica soot deposition:

| Siloxane                     | Si Delivery Rate (Si atoms per minute) | Amount of Silica Soot Deposited (grams) | Rate of Silica Soot Deposited (grams/minute) |
|------------------------------|--|---|--|
| Octamethylcyclotetrasiloxane | 3.62                                   | 1205                                    | 3.26   |
| Decamethylcyclopentasiloxane | 3.63                                   | 1186                                    | 3.21   |
| Hexamethyldisiloxane         | 2.64                                   | 855                                     | 2.31   |

(Powers Declaration ¶ 6). Thus, the polymethylcyclosiloxanes of the present invention achieve a substantially better rate of silica soot deposition than hexamethyldisiloxane.

An attempt was also made to utilize an atomic Si delivery rate of 3.62 grams of Si atoms per minute of hexamethyldisiloxane which was similar to the above atomic Si delivery rates of 3.62 and 3.63 grams of Si atoms per minute of octamethylcyclotetrasiloxane and decamethylcyclopentasiloxane, respectively (Powers Declaration ¶ 7). However, the burner flame became unstable, black carbon-laden soot was generated, and a non-uniformly shaped preform was produced (Id.). As a result, this run was terminated (Id.). Dr. Powers has extensive and impressive experience in fabricating fused silica glass and optical fibers (Powers Declaration ¶ 3). Based on his expertise in this field, he believes that silica deposition achieved with hexamethyldisiloxane is most fairly compared with silica deposition achieved with octamethylcyclotetrasiloxane and decamethylcyclopentasiloxane when similar volumetric flowrates are utilized for each (Powers Declaration ¶ 8). As the above data clearly demonstrates, the polymethylcyclosiloxanes of the present invention (e.g., octamethylcyclosiloxane and decamethylcyclopentasiloxane) achieve substantially higher levels and rates of soot deposition than linear siloxanes (e.g., hexamethyldisiloxane). Schwarz teaches that hexamethyldisiloxane is preferred over other disclosed alternatives, such as polymethylcyclosiloxanes. Therefore, one of ordinary skill in the art would have expected hexamethyldisiloxane to achieve better results to the extent, if at all, Schwarz was relevant to making a consolidated glass body (Powers Declaration ¶ 9). However, as shown by the above results, octamethylcyclotetrasiloxane (and decamethylcyclopentasiloxane) achieved substantially higher silica soot deposition rates than hexamethyldisiloxane (Id.). In view of Schwarz's disclosure of hexamethyldisiloxane as preferred, the results obtained in the above experiment are, in Dr. Powers' opinion, highly unexpected (Id.). Such unexpected results rebut any *prima facie* case of obviousness based on the combination of Miller, Schwarz, Hyde, and Kawaguchi. See In re DeBlauwe, supra.

The examiner has taken the position that the above data does not support a finding of unexpected results, because one of ordinary skill would expect trisiloxane to create 50% more silica than disiloxane, and pentasiloxane to create 150% more silica than disiloxane. The basis for this view is that trisiloxane has 50% more silicon atoms, and pentasiloxane has 150% more silicon atoms, than disiloxane. However, this view is unrealistic and ignores the significance of the results described

in the Powers Declaration. In particular, the values were calculated based on the Ideal Gas Law as opposed to any "real world" model. There is also no indication of how the examiner's proposed percentages relate to specific values in the Powers Declaration (at ¶¶5-8). In any event, the Powers Declaration demonstrates that the "rate of silica soot deposited" (measured in grams/minute) for polymethylcyclosiloxanes (as claimed in the present invention) is substantially higher than that of linear siloxanes. In view of Schwarz's express teaching that linear siloxanes are preferred over polymethylcyclosiloxanes for making pyrogenic silica, the results set forth in the Powers Declaration must be interpreted as unexpected.

Further, as shown in Example 4 and Figure 4 of the present application, the use of octamethylcyclotetrasiloxane has substantially improved deposition efficiency over  $\text{SiCl}_4$ . This is highly unexpected, particularly when one considers that Schwarz, in using such polycyclosiloxanes, does not suggest that the combustion product of this starting material can even form a deposit on a carrier, let alone deposit with greater efficiency. In response to this argument, the examiner has asserted that the unexpected results shown in Figure 4 of Example 4 are not commensurate with the scope of the claims, because Figure 4 only involves low production rates of silica from octamethylcyclosiloxanes ("OMCTS"). Once again, the examiner has ignored the significance of appellants' results. Example 4, and particularly Figure 4, shows that "OMCTS-based soot was found to deposit more efficiently than  $\text{SiCl}_4$ -based soot" (column 10, line 51 to column 11, line 1) and demonstrates "this difference as a function of the total amount of  $\text{SiO}_2$  produced at the burner for a specific blank size" (column 11, lines 2-4). Thus, in contrast with the use of  $\text{SiCl}_4$ , the use of OMCTS resulted in a favorable reduction in the quantity of particulate emissions (e.g., of HCl) while at the same time increasing the rate of production of silica (column 11, lines 4-7). In addition, the following passage from Example 4 shows that the relative increase in deposition efficiency using OMCTS, compared to  $\text{SiCl}_4$ , is consistent no matter what amount of starting material is used:

In optical waveguide production, deposition efficiency increases with increasing blank size. As deposition begins, collection deficiencies for  $\text{SiCl}_4$  frequently are less than 10%, whereas the use of OMCTS can yield initial deposition efficiencies up

to 25%. This factor of greater than twofold efficiency in deposition results in a corresponding increase in preform growth rate for equivalent rates of SiO<sub>2</sub> particles exiting the burner, and about a 20% by weight or more decrease in soot that has to be cleaned from the exhaust gases. (And in addition, of course, the use of OMCTS eliminates the costs involved in removing HCl from exhaust gases.)

(column 11, lines 13-24).

In discussing Figure 4, the examiner hypothesizes that the highest efficiency of the OMCTS and SiCl<sub>4</sub> precursors were nearly identical to one another, allegedly indicating an absence of unexpected results. However, there is no basis in Figure 4 for this speculative assertion. When the deposition efficiency values (indicated on the y-axis of Figure 4) are compared based on the amount of SiO<sub>2</sub> produced at the burner (x-axis of Figure 4), it is clear that OMCTS consistently has a significantly higher deposition efficiency value than SiCl<sub>4</sub>. Thus, claims 36-38 and 51-53, which are directed to the use of octamethylcyclosiloxane, are patentable of their own accord.

For all of these reasons, the obviousness rejection of claims 12, 13, 22, 33-39, 41-44, 46, 47, 49, and 51-53 over Miller in view of Schwarz, and, optionally, Hyde and/or Kawaguchi, is improper and should be withdrawn.

## IX. CONCLUSION

In view of the foregoing, it is clear that the rejection of the claims under 35 U.S.C. § 103(a) cannot be sustained. Accordingly, the final rejections should be reversed.

Respectfully submitted,

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## APPENDIX A

### In the Claims:

12. (Amended) [A method according to claim 7] In a method for making a non-porous body of high purity fused silica glass doped with at least one oxide dopant comprising the steps of:

(a) producing a gas stream containing a silicon-containing compound in vapor form capable of being converted through thermal decomposition with oxidation or flame hydrolysis to SiO<sub>2</sub> and a compound in vapor form capable of being converted through oxidation or flame hydrolysis to at least one member of the group consisting of P<sub>2</sub>O<sub>5</sub> and a metal oxide which has a metallic component selected from Group IA, IB, IIA, IIB, IIIA, IIIB, IVA, IVB, VA, and the rare earth series of the Periodic Table, wherein said compound in vapor form capable of being converted to at least one member of the group consisting of P<sub>2</sub>O<sub>5</sub> and a metal oxide which has a metallic component selected from Group IA, IB, IIA, IIB, IIIA, IIIB, IVA, IVB, V[B]A, and the rare earth series of the Periodic Table is a halide-free compound;

(b) passing said gas stream into the flame of a combustion burner to form amorphous particles of fused SiO<sub>2</sub> doped with an oxide dopant;

(c) depositing said amorphous particles onto a support; and

(d) either essentially simultaneously with said deposition or subsequently thereto consolidating said deposit of amorphous particles into a non-porous body; the improvement comprising utilizing as said silicon-containing compound in vapor form a halide-free polymethylcyclosiloxane, whereby no halide-containing vapors from said silicon-containing compound are emitted during the making of said non-porous body of high purity fused silica glass.

13. (Amended) In a method for making optical waveguide fibers of high purity fused silica through the outside vapor deposition process comprising the steps of:

- (a) producing a gas stream containing a silicon-containing compound in vapor form capable of being converted through thermal decomposition with oxidation or flame hydrolysis to  $\text{SiO}_2$ ;
- (b) passing said gas stream into the flame of a combustion burner to form amorphous particles of fused  $\text{SiO}_2$ ;
- (c) depositing said amorphous particles onto a mandrel;
- (d) consolidating said deposit of amorphous particles into a non-porous, transparent glass body; and
- (e) [and] drawing optical waveguide fiber from said body;  
the improvement comprising utilizing as said silicon-containing compound in vapor form a halide-free [polymethylsiloxane] polymethylcyclosiloxane, whereby no halide-containing vapors are emitted during the making of said optical waveguide fibers.

22. (Amended) [A method according to claim 17] In a method for making optical waveguide fibers of high purity fused silica glass doped with an oxide dopant comprising the steps of:

- (a) producing a gas stream containing a silicon-containing compound in vapor form capable of being converted through thermal decomposition with oxidation or flame hydrolysis to  $\text{SiO}_2$  and a compound in vapor form capable of being converted through oxidation or flame hydrolysis to at least one member of the group consisting of  $\text{P}_2\text{O}_5$  and a metal oxide which has a metallic component selected from Group IA, IB, IIA, IIB, IIIA, IIIB, IVA, IVB, VA, and the rare earth series of the Periodic Table;
- (b) passing said gas stream into the flame of a combustion burner to form amorphous particles of fused  $\text{SiO}_2$  doped with an oxide dopant, wherein said compound in vapor form capable of being converted to at least one member of the group consisting of  $\text{P}_2\text{O}_5$  and a metal oxide which has a metallic component selected

from Group IA, IB, IIA, IIB, IIIA, IIIB, IVA, IVB, VA, and the rare earth series of the Periodic Table is a halide-free compound;

(c) depositing said amorphous particles onto a mandrel;

(d) consolidating said deposit of amorphous particles into a non-porous transparent glass body; and

(e) drawing waveguide fiber from said body, the improvement comprising utilizing as said silicon-containing compound in vapor form a halide-free polymethylcyclosiloxane, whereby no halide-containing vapors from said silicon-containing compound are emitted during the making of said optical waveguide fibers.

33. A method according to claim 12, wherein said polymethylcyclosiloxane is selected from the group consisting of octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, hexamethylcyclotrisiloxane, and mixtures thereof.

34. A method according to claim 13, wherein said polymethylcyclosiloxane is selected from the group consisting of octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, hexamethylcyclotrisiloxane, and mixtures thereof.

35. A method according to claim 22, wherein said polymethylcyclosiloxane is selected from the group consisting of octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, hexamethylcyclotrisiloxane, and mixtures thereof.

36. A method according to claim 12, wherein said polymethylcyclosiloxane is octamethylcyclotetrasiloxane.

37. A method according to claim 13, wherein said polymethylcyclosiloxane is octamethylcyclotetrasiloxane.

38. A method according to claim 22, wherein said polymethylcyclosiloxane is octamethylcyclotetrasiloxane.

39. (Amended) In a method for making a non-porous body of high purity fused silica glass comprising the steps of:

(a) producing a gas stream containing a silicon-containing compound in vapor form capable of being converted through thermal decomposition with oxidation or flame hydrolysis to SiO<sub>2</sub>;

(b) passing said gas stream into the flame of a combustion burner to form amorphous particles of fused SiO<sub>2</sub>;

(c) depositing said amorphous particles onto a support; and

(d) either essentially simultaneously with said deposition or subsequently thereto consolidating said deposit of amorphous particles into a non-porous body;

the improvement comprising utilizing as said silicon-containing compound in vapor form, a halide-free polymethylcyclosiloxane selected from the group consisting of octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, hexamethylcyclotrisiloxane, and mixtures thereof, whereby no halide-containing vapors are emitted during the making of said non-porous body of high purity fused silica glass.

41. A method according to claim 39 wherein said gas stream is comprised of an inert gas.

42. A method according to claim 41 wherein said inert gas is nitrogen.

43. In a method for making a non-porous body of high purity fused silica glass doped with at least one oxide dopant comprising the steps of:

(a) producing a gas stream containing a silicon-containing compound in vapor form capable of being converted through thermal decomposition with oxidation or flame hydrolysis to SiO<sub>2</sub> and a compound in vapor form capable of being converted through oxidation or flame hydrolysis to at least one member of the group consisting of P<sub>2</sub>O<sub>5</sub> and a metal oxide which has a metallic component selected from Group IA, IB, IIA, IIB, IIIA, IIIB, IVA, IVB, VA, and the rare earth series of the Periodic Table;

(b) passing said gas stream into the flame of a combustion burner to form amorphous particles of fused SiO<sub>2</sub> doped with an oxide dopant;

(c) depositing said amorphous particles onto a support; and

(d) either essentially simultaneously with said deposition or subsequently thereto consolidating said deposit of amorphous particles into a non-porous body; the improvement comprising utilizing as said silicon-containing compound in vapor form a halide-free polymethylcyclosiloxane, whereby no halide-containing vapors from said silicon-containing compound are emitted during the making of said non-porous body of high fused silica glass.

44. A method according to claim 43 wherein said polymethylcyclosiloxane is selected from the group consisting of octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, hexamethylcyclotrisiloxane, and mixtures thereof.

46. In a method for making optical waveguide fibers of high purity fused silica glass doped with an oxide dopant comprising the steps of:

(a) producing a gas stream containing a silicon-containing compound in vapor form capable of being converted through thermal decomposition with oxidation or flame hydrolysis to SiO<sub>2</sub> and a compound in vapor form capable of being converted through oxidation or flame hydrolysis to at least one member of the group consisting of P<sub>2</sub>O<sub>5</sub> and a metal oxide which has a metallic component selected

from Group IA, IB, IIA, IIB, IIIA, IIIB, IVA, IVB, VA, and the rare earth series of the Periodic Table;

- (b) passing said gas stream into the flame of a combustion burner to form amorphous particles of fused SiO<sub>2</sub> doped with an oxide dopant;
- (c) depositing said amorphous particles onto a mandrel;
- (d) consolidating said deposit of amorphous particles into a non-porous transparent glass body; and
- (e) drawing waveguide fiber from said body; the improvement comprising utilizing as said silicon-containing compound in vapor form a halide-free polymethylcyclosiloxane, whereby no halide-containing vapors from said silicon-containing compound are emitted during the making of said optical waveguide fibers.

47. A method according to claim 46 wherein said polymethylcyclosiloxane is selected from the group consisting of octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, hexamethylcyclotrisiloxane, and mixtures thereof.

49. (Amended) In a method of making high purity fused silica glass through the outside vapor deposition process comprising the steps of:

- (a) producing a gas stream containing a silicon-containing compound in vapor form capable of being converted through thermal decomposition with oxidation or flame hydrolysis of SiO<sub>2</sub>;
- (b) passing said gas stream into the flame of a combustion burner to form amorphous particles of fused SiO<sub>2</sub>;
- (c) depositing said amorphous particles onto a mandrel; and
- (d) consolidating said deposit of amorphous particles into a non-porous, transparent glass body;

the improvement comprising utilizing as said silicon-containing compound in vapor form a halide-free polymethylcyclosiloxane selected from the group consisting of octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, hexamethylcyclotrisiloxane, and mixtures thereof, whereby no halide-containing

vapors from said silicon-containing compound are emitted during the making of said high purity fused silica glass.

51. A method according to claim 49, wherein said polymethylcyclosiloxane is octamethylcyclotetrasiloxane.

52. A method according to claim 39, wherein said polymethylcyclosiloxane is octamethylcyclotetrasiloxane.

53. A method according to claim 43, wherein said polymethylcyclosiloxane is octamethylcyclotetrasiloxane.